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EXOTHERMIC BRAZING SPECIMENS E FILE COPY

FINAL REPORT

Covering Period 22 June 1967 to 31 December 1970

Prepared for
National Aeronautics and Space Administration
Huntsville, Alabama

WHITTAKER CORPORATION Research & Development Division 3540 Aero Court San Diego, California 92123

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FOREWORD

This final report was prepared by Whittaker Corporation, Research and Development Division, San Diego, California under Contract No. NAS-8-20829, Control No. DCN-1-7-30-12665 for the George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Huntsville, Alabama. Mr. J. C. McCaig was acting as Project Engineer.

This report summarizes the three-phase development and manufacture of exothermic brazing packages covering the period from 22 June 1967 to 31 December 1970.

Mr. Frank J. Filippi of Whittaker was Program Manager for Phase I, assisted by Mr. F. A. Barr, both reporting to Mr. B. L. Duft, Manager, Engineering Department. Mr. Stanley Rodney, Physicist, was Program Manager for Phases II and III and directed the laboratory effort on the program, reporting to Dr. B. R. Garrett, Manager of Whittaker's Product and Process Development Department.

SUMMARY

It was the purpose of this program to design, optimize and fabricate exothermic braze units for repair and assembly of stainless steel materials on space missions. The units had to be lightweight, compact, compatible with space environments and easily handled by astronauts during space operations. All objectives of the program were successfully completed including fabrication of final flight hardware. Well over 200 test units were manufactured and evaluated during the development phase.

Final design of the braze packages employed Whittaker exotherm system No. 34, a mixture of metals and metal oxides, to produce the necessary 650 ± 30 calories/gram for two-way braze alloy flow. Ignition was accomplished using a 24-volt battery and an igniter consisting of a coiled 0.008" tungsten wire bridge embedded in pyrotechnic 116/17 Ti. 3/4" stainless steel tubing was joined successfully with the joints being essentially void-free.

A reliability level of 95% was desired in the final exothermic braze package. To accomplish this level of performance a rigid inspection and assembly procedure was established. Final manufacturing procedures required that the braze units be packaged in polyethylene under nitrogen and radiographically inspected and approved prior to shipment.

During the design and optimization stages Marshall Space Flight Center evaluated and tested each modification. The final design evolving from this development work passed performance requirements and thirty-two (32) flight hardware units were fabricated for evaluation under actual space conditions.

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INTRODUCTION

As the United States space effort develops into more complex missions the need for improved, lightweight joining methods for in-space repair and assembly procedures becomes increasingly important.

This report summarizes a program undertaken by Whittaker Corporation to design and fabricate exothermic braze specimens which are capable of operating under gravity and zero gravity conditions in a 10^{-6} to 10^{-8} Torr vacuum and providing for two-way braze alloy flow. The program consisted of three phases:

Phase I Design, optimization and exploratory evaluation of exothermic braze specimens.

Phase II Qualification testing of exothermic units for flights.

Phase III Flight hardware.

Specimens were to be prepared during Phase I and supplied for experimental tests to be performed at the Marshall Space Flight Center laboratory. In Phase II design of the units was to be finalized and additional braze packages provided for testing by MSFC. Flight hardware was to be prepared in Phase III for actual evaluation by astronauts in outer space.

The term exothermic brazing refers to a process whereby the heat required to melt or flow a brazing filler metal is generated by a solid-state chemical reaction between one or more active metals and reducible metal oxides. This differs from the well-known thermite welding process in that only the heat generated by the chemical reaction is used and the bonded area is not contaminated in any way from the by-products of the reaction. The process, therefore, differs from other brazing methods only in the source of heat.

Exothermic mixtures can be ignited into a self-propagating reaction by passing current from a low-voltage battery through a fine tungsten wire heating the wire to the ignition temperature of the surrounding exotherm. The rate of heating and the total usable heat produced per unit mass of exothermic material can be determined and controlled over a wide range. Control is established by chemical compounding and by matching the exothermic source to the heat sink of the object to be heated.

Materials may be added to the exothermic heat source to control such properties as specific heat, thermal conductivity, emissivity of the mix, the character of reaction products, and the thermal-pulse shape.

An integrated design concept was employed for developing the exothermic braze packages require for Phase I. These units were designed for specific

types and sizes of tubing and contained the coupling, brazing filler material, exotherm mixture, and package insulation. Figures 1 and 14 depict a typical exothermic brazing package developed during this phase.

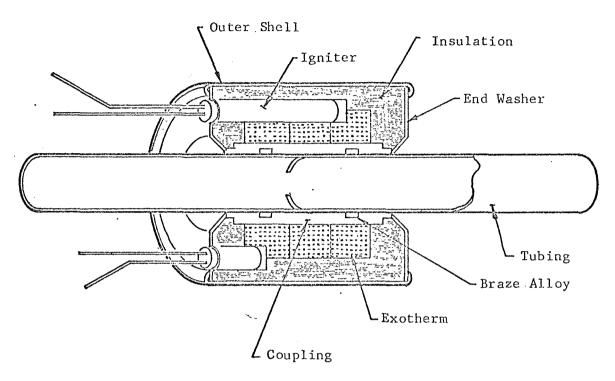
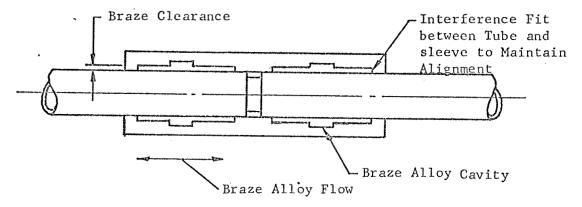


Figure 1. Cross Section of Exothermic Braze Unit

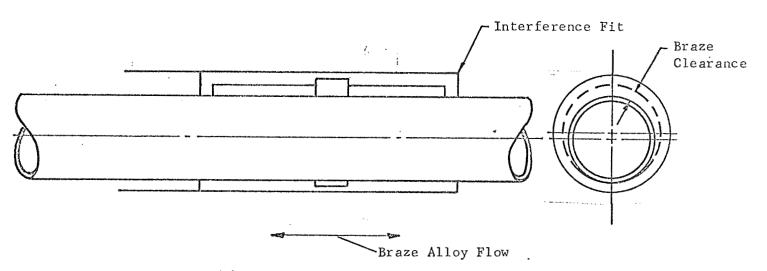
The exothermic braze packages initially developed in Phase I were for the following size of stainless steel tubing:

Tube Outside	Approximate Tube Wall
Diameter, Meter (Inch)	Thickness, Meter (Inch)
0.00635 (0.250)	0.000508 (0.020)
0.01905 (0.750)	0.001245 (0.049)

Design and development of the exothermic brazing packages were based on requirements shown in sketches SK# MR&TSK-1296, concentric braze clearance joints, and SK# MR&TSK-1297, eccentric braze clearance joints. Both sketches are reproduced here as Figure 2. The tubing and sleeve (coupling) material used in this investigation were fabricated from 347 stainless steel, while the braze alloy specified was AWS A5.8-62 Class B Ag-8a filler material (71.8% silver, 28% copper, 0.2% lithium). Approximately 130 exothermic braze units of the following sizes were to be prepared during Phase I:



(a) Concentric Braze Clearance Joints



(b) Eccentric Braze Clearance Joints

Figure 2. Braze Clearance Joints. The braze alloy will flow per sketches. Interference fit of sleeve to tube will maintain tube alignment and braze clearance during exothermic thermal pulse. Variations in braze clearance will be explored for characteristics and range of braze alloy flow to determine relative relationship between gravity and non-gravity performance (From SK #M&FTSK-1296 and SK #M>SK-1297). Phase I.

50 each: 0.00635 meter (1/4 inch) concentric 50 each: 0.01905 meter (3/4 inch) concentric 30 each: 0.01905 meter (3/4 inch) eccentric

These units were to be evaluated by MSFC and, utilizing results of their tests, additional test units were to be designed and fabricated. Final design of the exothermic braze units was to be established in Phase II and test packages were to be supplied to Marshall Space Flight Center for more extensive testing and for use in the training of astronauts. In Phase III additional exothermic braze packages were to be fabricated, radiographically analyzed and supplied to MSFC for evaluation in actual welding experiments in outer space. All phases of the program were successfully completed.

Design of the braze units made in Phase I was based upon experimental tests conducted at MSFC and were fabricated to meet requirements shown in sketches SK #M&FTSK-1296 and SK #M>SK-1297. Experiments were conducted with various braze alloys but none were found superior to AWS A5.8-62 Class B Ag-8a and it was used in all of the final exothermic braze packages. The tubing and sleeve (coupling) material used in this investigation was fabricated from 347 stainless steel as furnished by MSFC. Approximately 90 units, .01913" meter I.D. (0.754"), were made during Phase II and Phase III with the last 75 being essentially identical. Four of the 75 braze packages were designed with a slightly larger inside diameter, 0.01937 meter (0.7660), couplings.

A typical 347 S.S. tubular joint made with a 0.01905 meter (3/4") diameter exothermic braze unit using the silver-copper-lithium braze alloy is shown in Figure 3.

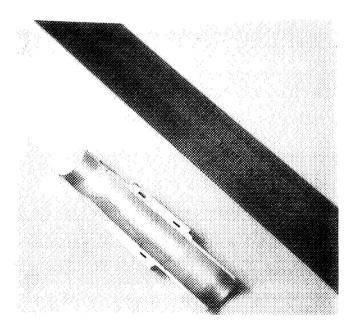


Figure 3. Typical 347 S.S. tubular joint made with a 0.01905 meter (3/4") diameter exothermic braze unit using the silver-copper-lithium braze alloy.

Based upon the results of these brazing experiments, well bonded joints can be made with tubular stainless steel. The joining of stainless steel and other metal tubing in diameters up to 0.1522 meters (6 inches) appears feasible.

TECHNICAL DISCUSSION

To design and fabricate exothermic tube-brazing packages for space use with an overall performance reliability approaching 99% requires careful control in the manufacture of all components and critical evaluation of the braze units under space-simulated conditions. The successful completion of this program was due in large measure to the realization of these development goals.

Exothermic brazing units suitable for space demands that the following general requirements be met:

- 1. Operational capability under gravity and zero gravity and hard vacuum (10^{-8} to 10^{-6} Torr) conditions.
- 2. Overall performance reliability of 99%, with a 95% confidence level.
- 3. Supply controlled, uniform heat sufficient to braze stainless steel tubing with minimum voids.
- 4. Ignition of exothermic materials using a 24-26 volt battery power source.
- 5. Easy application of braze units under space conditions.
- 6. Containment of all chemical by-products from exothermic reaction.
- 7. Non-contaminating to controlled space module environment.

The design and fabrication of each component in the exothermic brazing package and the assembly procedures are discussed in the following sections.

Coupling Design

Considerable study preceded the design of the couplings. Coupling designs were based on Type 347 CRES fittings in the annealed condition. The specification of the annealed condition requirement had both a mechanical and metallurgical design basis. Mechanically, annealing would minimize braze gap changes and general distortion of the coupling, a factor of primary importance to the Phase I design and development work. Metallurgically, the annealed stainless steel would minimize or prevent the occurrence of intergranular braze alloy penetration in a properly adjusted brazing cycle. Emphasis was placed on this design consideration, since a previous program concerning vacuum brazing of austenitic stainless steel tubing revealed occasional cracking and brazed alloy penetration of the brazed joints.

Stress corrosion cracking of this type has been reported for austenitic stainless steels in contact with molten silver-base filler alloys. These stresses can arise from:

- 1. Residual stress (the couplings and tubing can be in a work-hardened condition through a sizing operation such as swaging) and also stresses induced by post-thermal heat treatment (solution hardening, etc.).
- 2. Thermal stresses caused by the heating process (exothermic brazing applies localized heat at a rapid rate).

This high-temperature stress-corrosion-cracking phenomenon can be minimized by:

- 1. Annealing the components to be brazed.
- 2. Using longer and heavier walled couplings to cause slower heating up of the tubing and thereby produce a decreased thermal gradient in the tubing.

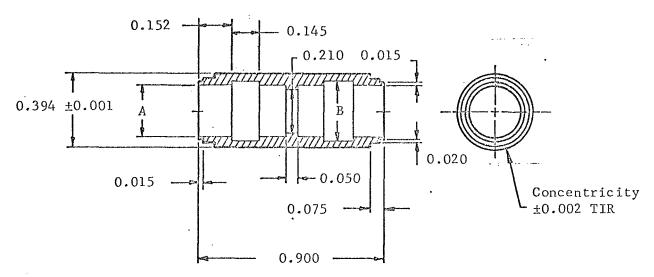
Figures 4 and 5 show the 0.00635 meter (1/4") and 0.01905 meter (3/4") coupling designs which provided for the various coupling-to-tubing clearances desired under this phase. Positioning was to be maintained in practice by use of longitudinal wires of correct size located peripherally approximately 120° apart.

Coupling wall thicknesses were sized to the minimum cross section. The outer diameters of the couplings were held constant to minimize exotherm compacting die costs. Coupling lengths were established in keeping with previous burst test experience, and amount of brazing alloy (groove depth and width) was calculated. (Calculations regarding the design of these couplings are listed in Appendix A).

The steps on the outer diameter of the couplings served two purposes: they minimized stress concentration at the outboard end, and the outermost step was used for positioning the package end closures.

During the preparation of the braze packages, subsequently discussed, the tubing stop on the couplings was eliminated by NASA in order to facilitate the Phase I work. Consequently, a slotted one-piece tube was employed to simulate the cut. Therefore, it should be noted that the original braze length safety factors (shown in Appendix A) of 8.75 and 7.15 for the 0.00635 meter (1/4") and 0.01905 meter (3/4") couplings were increased to 9.5 and 7.6, respectively, by removal of the center stop making more braze area available.

After evaluating the Phase I braze packages at Marshall Space Flight Center it was apparent that in order to achieve a high level of reliability (approximately 95%) the rotation of the braze unit on the sleeve must be

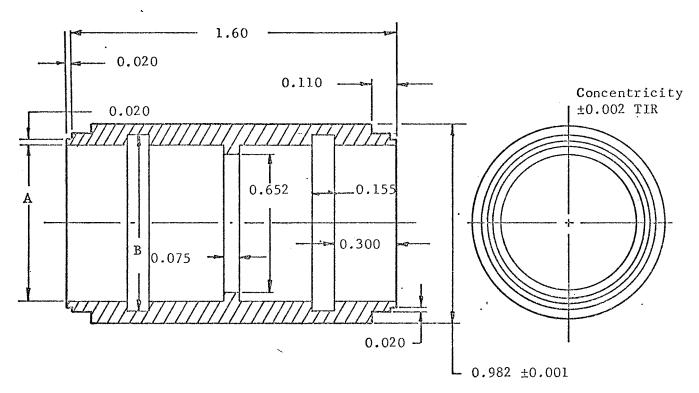


Scale: 2:1 Material: 347 stainless steel, fully annealed All dimensions ± 0.002 unless otherwise stated All outside corners broken — All inside 0.005 - 0.010 radius

No.	A(±0.00025)	B +0.003 -0.001
1	0.253	0.312 .
2	0.256	0.312
3	0.262	0.312
4	0.274	0.344

0.250-in. Tube OD assumed

Figure 4. 0.00635 Meter (0.250") Coupling Phase I



Scale: 2:1
Material: 347 stainless steel, fully annealed
All dimensions ±0.002 unless otherwise stated
All outside corners broken.— All inside 0.005 - 0.010 radius

No.	A(±0.00025)	B(+0.003) -0.001)
1	0.754	0.846
2	0.758	0.846
3	0.766	0.846
4	0.770	0.922
.5	0.782	0.922

0.750-in. tube OD assumed

Figure 5. 0.01905 Meter (0.750") Coupling Phase I

eliminated. This was accomplished in Phase II by machining a hexagonal configuration on each end of the sleeve (Figure 6). Housing end plates were then machined to fit snugly over the hexagonal sleeve ends to prevent rotation. This design, while eliminating the steps on the outer diameter of the couplings, effectively stabilized the entire assembly. The final braze package design is shown in Figure 6.

Exotherm Material

The heart of the exothermic brazing unit is the exothermic reactant mixture. These reactants are blends of various reactive metals and reducible oxide powders. Selection of the exotherm material for this program was based on past knowledge and on information from a closely related program in which Whittaker supplied exothermic braze packages.*

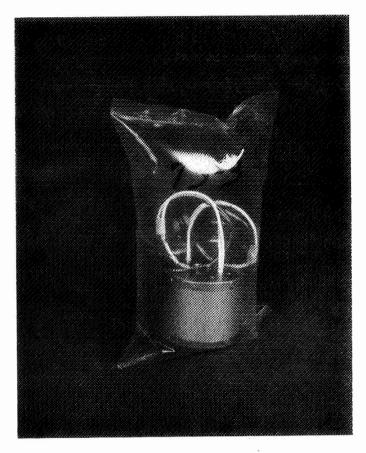
Exotherm mixtures containing boron and vanadium pentoxide were found to be necessary in order to ignite and propagate in a vacuum. Consideration was also given to the heat required by the braze alloy which was composed of 72 weight percent silver + 28 weight percent copper and had a melting and flow temperature of 779°C (1435°F). The exothermic material was also selected on the basis of providing the largest amount of heat with the least weight and volume. Further consideration was given to using an exotherm which retained its basic compacted shape after ignition. Thus, uniform heating around the entire coupling periphery would be realized under various attitudes and conditions of gravity.

In Phase I preliminary evaluation of exotherm materials in initial 0.00635 meter (1/4") and 0.01905 meter (3/4") packages led to the selection of Whittaker's exothermic system #36. This system was prepared from the following powdered raw materials:

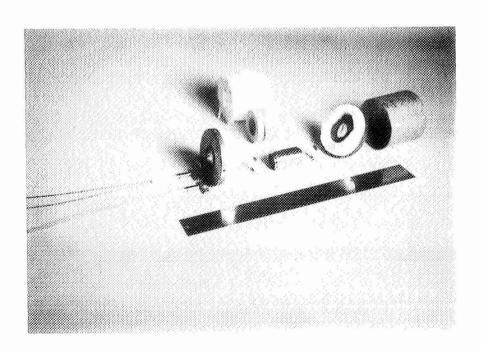
Vanadium pentoxide
Titanium dioxide
Nickel oxide
Manganous dioxide
Manganous oxide
Aluminum metal
Magnesium metal
Boron metal

In order to ensure reliability and uniformity throughout the program, a single master batch of the above system was mixed and blended, then tested against existing Whittaker specifications.

^{*}H. T. Mischel and P. J. Valdex, "Testing and Evaluation of Exothermal Braze Specimens," Contract No. NAS 8-11282, Marshall Space Flight Center, September 1966.



(a) Completed Package under Nitrogen



(b) Exploded View - Final Design

Figure 6. Final Exothermic Braze Package Assembly

Following the evaluation of most of the braze packages prepared in Phase I it was found that the uniformity of heat distribution and reproducibility from one unit to another had to be upgraded. Marshall Space Flight Center also found that after several months storage a high percentage of the braze units failed to fire, possibly due to a shelf life limitation with the exotherm system. To correct these deficiencies it was decided to use Whittaker's exotherm system #34 in Phase II since no storage or firing difficulties had been detected with this exotherm. System #34 had an ignition temperature of $1104 \pm 10\,^{\circ}\text{C}$ ($2020 \pm 50\,^{\circ}\text{F}$) with a caloric output of 650 ± 30 cal/gram. No manufacturing difficulties were encountered with exotherm system #34 and a single master batch was used in making the last 75 exothermic braze packages for Phase II and Phase III. Numerous tests conducted by MSFC indicated that system #34 performed quite satisfactorily. A 60-gram loading of exotherm system #34 was used in fabricating the final 75 0.01905 meter (3/4") diameter braze packages.

Compaction of the exothermic material for use in braze packages was accomplished by cold pressing at $6.89 \times 10^7 \text{ N/m}^2$ (10,000 psi) in hardened steel dies. Exothermic rings of 0.0253 meter (0.997") ID x 0.04445 meter (1.750") OD were pressed for 0.01905 meter (3/4") packages, and 0.01016 meter (0.400") ID x 0.01905 meter (0.750") OD for the 0.00635 meter (1/4") units. The selected exotherm system in compacted form is safe to handle and store and difficult to ignite without an electrical igniter.

Exotherm rings were pressed to various thicknesses to determine optimum amounts required for void-free brazing of the various tube sizes.

Igniter Development

As previously mentioned, the exotherm reactants had to be brought to a temperature sufficient enough for the reaction to initiate and proceed simultaneously. Only a small area required heating in order to propagate the reaction. This permits ignition to take place from a small hot-wire bridge requiring minimum power.

To assure reliability, a redundant ignition system (two igniters in parallel) was required for the exothermic braze packages. Their position in the package is discussed in a subsequent section.

Figure 7 depicts the cross section of the igniter designed to operate with a 24-volt DC power source. Basically, the igniter consisted of an 8-mil tungsten wire bridge embedded in 18-gauge un-insulated copper lead wire (Mil-W-3861/5) for reacting the igniter exotherm. The igniter exotherm employed has a quick heat pulse and is hot enough to initiate reaction of the main exotherm rings.

The igniter was made up of a thin-wall stainless tube, solid copper lead wires, a tungsten bridge wire, a ceramic thermal and electric insulator, and an igniter exotherm.

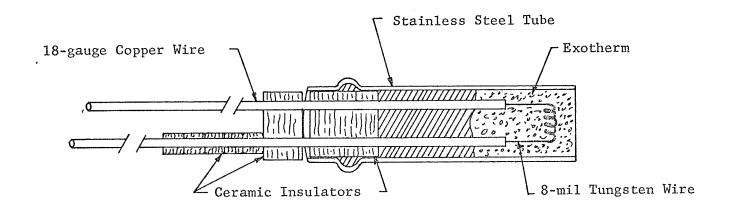


Figure 7. Igniter Cross Section

The igniter tube was cut to length and crimped to hold its position in the housing and also to lock the ceramic insulator in place.

The two 18-gauge copper lead wires were cut to length and inserted in an embedding tool which formed the copper wire around the 8-mil tungsten bridge wire. The wire assembly was then bent to form a U-shaped tungsten bridge at one end of the parallel copper lead wires, and, in turn, was placed on a positioning fixture. After conducting a number of tests early in Phase II it was decided to coil the tungsten bridge wire (as shown in Figure 7) in order to achieve more uniform and reliable ignition of the 116/17 Ti pyrotechnic. A crimped stainless sleeve was placed over the positioned leads, and the ceramic insulation in paste form was metered into the sleeve, leaving space above the bridge for the exotherm charge.

After the ceramic had cured, the unit was removed from the fixture, The ceramic now held the wires in position and isolated them from the stainless sleeve. Units were tested for electrical continuity and for electrical leakage to the metal tube before filling the cavity with igniter exotherm.

This type of igniter eliminated any plastic insulation and the associated undesirable smoking. Since the igniter was a single unit, it could be readily tested for electrical continuity before and after assembly into the braze package without disturbing the wiring or igniter exotherm.

Major efforts with the igniter unit consisted of obtaining a ceramic castable or potting compound suitable for electrically insulating the copper lead wires from each other and the outer shell. A high service temperature ceramic cement, Sauereisen No. 8, was selected. Other ceramic castables were tried but results were unsatisfactory, primarily because the difference in thermal expansion between the stainless tube and the ceramic caused cracks and broke the bond during curing of the ceramic.

Sauereisen No. 8 exhibits a room temperature chemical action cure and, after curing, has excellent thermal shock resistance. No igniter failures have been observed when this ceramic insulation has been used.

A safety test was performed on a loaded igniter to determine if premature ignition of its exotherm charge could take place during the connecting operations (e.g., soldering of the igniter lead wires to the power source circuit) as a result of thermal conduction through the leads into the igniter bridge. The igniter lead wire had a thermocouple attached to it a distance of 0.00318 meter (1/8") from the igniter body end from which the lead wires exit. A 200-watt soldering gun was placed across the igniter lead wires at a distance of 0.00635 meter (1/4") from the igniter body. Power was applied until no increase in lead wire temperature was noted. The maximum temperature reached was 298°C (569°F); the wire was kept at this temperature for 2 minutes. Ignition of the igniter charge did not take place.

It is believed that this test approximates a worst condition as applied to a normal soldering operation. A normal soldering operation as applied to this case would:

- 1. Take place within several seconds.
- 2. Occur at a temperature about 100°F lower than the test maximum.
- 3. Be made at a distance from the igniter body five to nine times that of the test heat source point.

A heat sink could be interposed between the point of connection and the igniter body if required during circuit assembly; although this test would appear to indicate that this action is unnecessary.

The above-described test was run under ambient laboratory conditions, since it was assumed that the assembly operation would be undertaken under these conditions.

Originally glass sleeving insulation was to be used on the igniter wire and was to be anchored firmly in the Saureisen ceramic cement. Preliminary tests at WRD and preparation of a few igniter test units indicated that it would be risky to use this type insulation when trying to achieve a 95% confidence level. After discussions with MSFC it was decided to use ceramic ball and socket type insulation. These small insulators were approximately 0.002794 meter (0.110") long x 0.002794 meter (0.110") OD x 0.001422 meter (0.056") ID, were strong and resistant to breakage and the igniter wires could be bent in tight radii without difficulty.

Package Development

The term <u>package</u> is applied to the complete integral unit comprising the tubing coupling, filler alloy, heat source, insulation, and igniter. In order to achieve a high level of reliability it is necessary to carefully check each component of the package and maintain tight quality control. The Inspection Procedure followed in the preparation and assembly of each component into the final design of exothermic braze packages for this program is shown in Appendix B. All of the final 75 braze units were assembled in accordance with this procedure. Design details and development of the various components is discussed below.

From experience gained through past programs concerned with exothermic heat source development for brazing applications, it has become apparent that the insulative portion is as critical as the components it houses. The insulation must be able to contain and arrest the high reaction temperatures without producing smoke, flames, or water of hydration. This requirement eliminates products that contain organic binders to make them formable, machinable, or physically stronger. However, binder reinforced ceramic fiber boards and even asbestos board (Johns-Mansville machinable Marinite 36) have been used where some smoke is not objectionable.

The most satisfactory material is a ceramic fibrous material, Fiberfrax, manufactured by Carborundum in blanket and felt form. Basically, the blanket and felt are made from alumina silicate fibers characterized by a melting point of about 3300°F. Extremely low thermal conductivity is one of the features of this insulation, with values ranging from less than 0.072 to 0.433 joule/m sec K° (1/2 to 3 Btu/hr ft² $^{\circ}$ F/in.). Although the exotherm reaction actually melts the Fiberfrax insulation where they come in contact with one another, the deterioration is very shallow and the remaining insulation mass effectively contains the reaction. The fibrous blanket and felt insulation does not produce any volatiles and tends to filter or condense any gaseous reaction products from the exotherm material.

Another material investigated during this phase was vacuum-drawn Fiberfrax board. It was hoped that this material could be cut into washers and end pieces to facilitate package assembly, since fewer parts would have to be made. However, the strength and texture of the board (hard surfaces but soft interior) did not permit satisfactory cutting of thick parts. Therefore, Fiberfrax blanket and felt were selected.

To facilitate construction, the insulation required was obtained by cutting the blanket and felt material into a definite number of washers for use in each position of the package. Previously, the insulation material had been packed around the exotherm as a batting.

In Phase I the outer housing of the package was fabricated from aluminum tubing cut to size and crimped at each end to hold and position the stainless steel end washers. Thin steel end washers were employed as heat shields to contain the heat loss through the porous ceramic insulation.

In this case, the end washers were formed to allow the exotherm rings to be placed farther outboard on the coupling for the two-way alloy flow and still allow a thick enough insulation layer on the ends. This arrangement is shown in Figure 1. However, testing of the Phase I units showed that excessive movement of the braze package around the sleeve may have been the cause of several misfirings. For this reason it was decided in Phase II to use a formed end plate with a hexagonal center hole which fit tightly on the hexagonal ends of the coupling. End plates were riveted to the outer stainless steel housing as shown in Figure 6. All 75 of the last 90 units produced in Phase II and Phase III were fabricated in this fashion.

All development screen testing of packages was performed in a vacuum chamber at a pressure of 3 \times 10⁻⁶ mm Hg or less. Figure 8 shows a typical test package while Figure 9 depicts the overall view of the screen testing arrangement. Ignition current was supplied for only the first few experiments by a battery. This was replaced by an adjustable DC power source, shown to the left on Figure 8. Thermocouple wires ran through the chamber walls, as was the case for ignition wiring through Conax connectors. The chamber had two glass ports for viewing the experiments.

The amount of exotherm (weight) required for each size unit was determined by using past information to obtain an initial weight fix and then empirically adjusting to obtain two-way alloy flow. These experiments as well as those performed on other areas of package development used tubing 0.1016 meter to 0.1524 meter (4" to 6") long to simulate the heat capacity of infinite or practical use length sections. The specimens were allowed to cool to at least below the surface oxidation temperature of the base metal before removal from the chamber.

Quick determinations were made of the completeness of the braze by sawing the coupling lengthwise and peeling the coupling from the tube. Figure 10 illustrates a typical peeled coupling, in this case showing complete brazing and two-way alloy flow for a 0.01905 meter (3/4") fitting.

The inside diameter of the pressed exotherm ring was held constant, based on the outside diameter measurement of the coupling plus a working clearance. The outer diameter of the exotherm ring was variable but since a hardened tool steel die was required for any size change, efforts were made to employ the thickness (height) of the rings as the limiting variable. Rings were then readily adjusted by changing the amount of material loaded in the die but were limited by the length of the coupling.

For instance the 0.00635 meter (1/4") coupling needed 7.5 grams (three rings, 2.5 grams each) for a complete braze using a ring 0.01016 meter (0.400") ID and 0.01778 meter (0.700") OD. In this case the exotherm rings covered the entire length of the coupling and the braze was complete but marginal. Therefore a new die was obtained with the same inside diameter but an outside diameter of 0.01905 meter (0.750"). This size change allowed 9 grams (three rings, 3 grams each) to be positioned over the same length and insured ample heat.

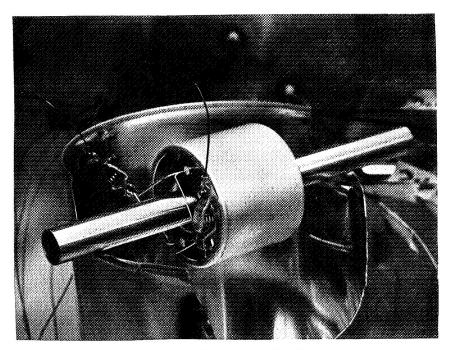


Figure 8. Test Package

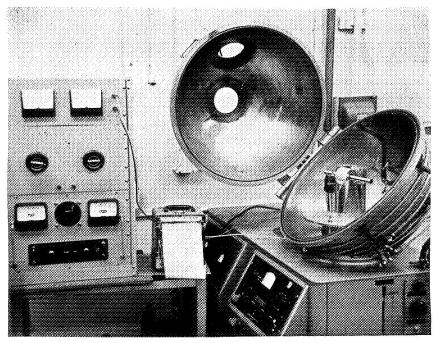


Figure 9. Test Arrangement for Braze Packages

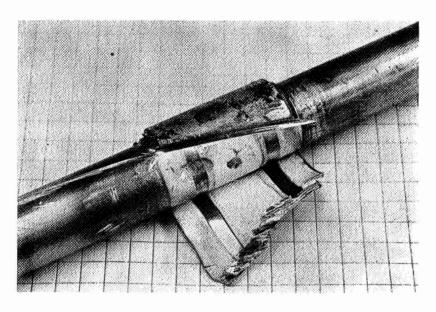


Figure 10. Peeled Exothermic Braze Specimen

The 0.01905 meter (3/4") unit was first tried using 72 grams of exotherm system #36 (three rings, 24 grams each). This proved more than necessary, as did 60 grams (three rings, 20 grams each). Fifty grams total made a satisfactory braze but the final units prepared for Phase I contained 54 grams total (three rings, 18 grams each) to insure ample heat. In Phase II it was determined that exotherm system #34 was more reliable and provided more uniform heat distribution. Optimum total exotherm weight was found to be 60 grams for this size braze unit and was used in the fabrication of all remaining units.

GFE (Government Furnished Equipment) tubing was supplied by NASA to be included with each individual prepared package. The tubing was supplied in the following sizes:

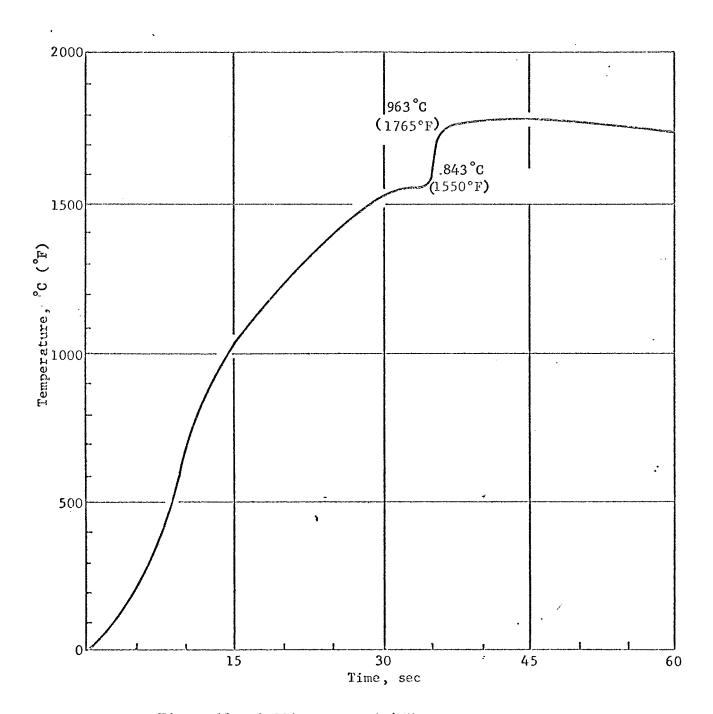
1/4-inch OD Tubing, 0.020-inch wall thickness, 4-inch length 3/4-inch OD Tubing, 0.049-inch wall thickness, 4-inch length

In Phase I the GFE 1/4-inch couplings were supplied only for the No. 1, 3 and 4 items listed in Figure 4. The 3/4-inch couplings were supplied only for the No. 1, 2, 3, and 4 items listed in Figure 5.

Preparation of the 1/4-inch units was evenly distributed using the No. 1, 3, and 4 couplings. This allowed 1/4-inch packages to be submitted to NASA for their evaluation having coupling-to-tubing clearances of 2, 6, and 12 mils respectively. In like manner the 3/4-inch concentric packages were prepared from the No. 1, 2, 3, and 4 couplings which provided for respective coupling-to-tubing clearances of 2, 4, 8, and 10 mils. The 3/4-inch eccentric units were fabricated using the No. 4-3/4-inch coupling which provided for various clearances up to a maximum of 20 mils.

Braze joint temperature profiles were determined during the various experiments by monitoring with chromel-alumel thermocouples and a Varian G-11 recorder. Thermocouples were attached by spot welding to the outside of the tube as close to the coupling as possible, about 0.001588 meter (1/16"). After finding the temperatures in a no braze and a braze situation it was decided to locate the thermocouple on the inside of the tube so that the temperature could be recorded in the braze area. The thermocouple was spot welded about 0.001588 meter (1/16") from the end of the tube and the tubes were butted in the center of the fitting.

The temperature of the tubing during the solidus to liquidus transformation of the alloy can easily be detected on the recorder chart. The required heat of fusion causes the temperature to level off, but when the alloy melts and contacts the tubing, direct conduction can take place between the heated fitting and the tube. This causes an abrupt change in the curve (see Figure 11). The temperature of the tube is between 843 $^{\circ}$ C (1550 $^{\circ}$ F) and 860 $^{\circ}$ C (1590 $^{\circ}$ F) (different specimens) just before alloy conduction.



0.00635 meter (1/4") Coupling Temperature Profile (Typical) Figure 11.

During testing of the 0.00635 meter (1/4") units and temperature of 963°C (1765°F) brazed inboard only, a high of 994°C (1822°F) brazed inboard plus about 50% of the outboard area. Complete two-way alloy flow was observed when the peak tubing temperature measured 1010°C (1850°F).

In like manner temperature profiles were determined for the 0.01905 meter (3/4") development packages. Temperature readings using 50 grams of exotherm charge showed that the braze alloy melted when the temperature was 783°C (1442°F). Complete brazing was accomplished by providing for a temperature of 916°C (1680°F).

To determine if the precautions taken to prevent stress corrosion embrittlement of the brazed coupling the tube had been effective, a metallographic survey consisting of a microscopic study of the microstructure in the braze area and a microhardness survey was accomplished. The specimen used for this survey was a 0.01905 meter (3/4") brazed joint in which the charge of 54 grams of exotherm system #36 found in the screening test to be optimum for this size was used. A section of the brazed joint was removed, polished and electrolytically etched in a 9% oxalic acid solution for one minute at 6 volts to reveal the general structure. The braze alloy is darkened by this etchant.

Examination of Figure 12 shows that no intergranular penetration of the base metal (either coupling or tubing) occurred. The presence of a braze alloy diffusion zone as depicted in the microstructure was an indication of good wetting and measured approximately 0.0003 inch at its widest point.

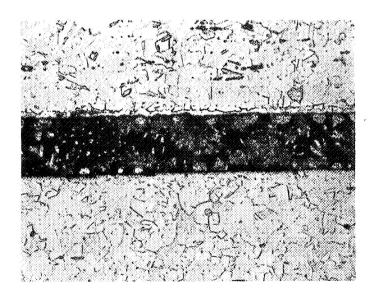


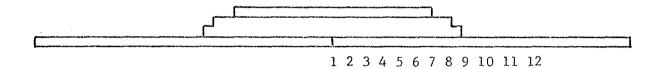
Figure 12. Photomicrograph of Braze Joint Interface (Magnification 266X)

The microhardness profile displayed in Figure 13 was made on the same polished section described above, prior to being etched. A Leitz microhardness tester was used with a 300-gram load. Readings were taken every 0.00254 meter (0.1") on both tube and coupling from the center of coupling to that portion of tubing virtually unaffected by the braze cycle.

The only apparent metallurgical effect of the brazing cycle on both coupling and tube was further annealing of the material.

Evaluation of Phase I exothermic braze units at Marshall Space Flight Center revealed a number of braze package design factors that had to be modified and improved. Some of the improvements included in the package produced during Phases II and III were:

- 1. Braze filler flow was changed to provide joints having less than 10% void area when the sleeve fit is optimum (0.004" clearance diameter).
- 2. Excess filler material was prevented from flowing through the joint and depositing on the inside of the tube.
- 3. Reproducibility and uniformity of heat distribution were improved with peak temperature variations minimized from one unit to another. Exotherm system #34 was used in place of exotherm system #36.
- 5. Perforations in the package housing were eliminated.
- 6. Friable material outside the exothermic braze package was eliminated.
- 7. Rotation of the exothermic braze package on the sleeve was eliminated by redesigning and locking the end washers in position with the sleeve.
- 8. Igniter lead wires were shown to be compatible with heat sinking provisions of the experimental package.
- 9. More uniform ignition rates were achieved by using a coiled tungsten igniter wire in the pyrotechnic.
- 10. Igniters were re-positioned to provide better alignment and were anchored firmly to the end plate with retainers.
- 11. Ball and socket ceramic insulators were used as external insulation on the igniter wires to replace less durable glass sleeving.
- 12. Completed braze packages were packaged in polyethylene bags under nitrogen.



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Coupling
                                                       Tube
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                                                                  171
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                                                       2. HV
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      AIR = HV
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                                                      12. HV
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                                                   B. AIR
                                                               HV = 187 = B 90
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NOTE:

- (1) Load = 300 grams
- (2) Each Reading represents 0.1 inch traverse

Figure 13. Vicker's Micro-Hardness Profile of Stainless Steel Brazement

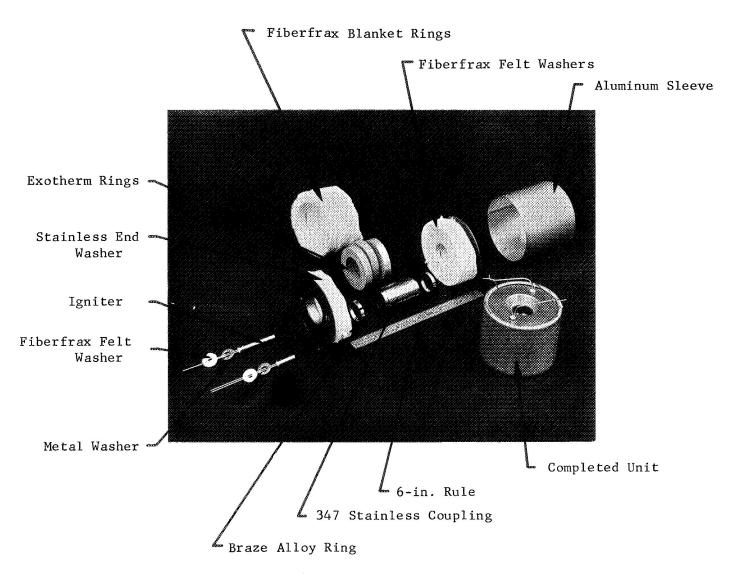


Figure 14. Phase I Exothermic Brazing Package

The last 75 exothermic braze packages produced for this program included all of the above improvements.

Inspection Procedure and Quality Control:

In order to achieve the highest level of reliability the fabrication of all exothermic braze packages was carefully monitored. An inspection and assembly procedure was established that required frequent testing or monitoring at several stages of fabrication. Exothermic Braze Package Inspection Procedure is appended as Appendix B. Included in this procedure are:

- 1. A breakdown of components comprising a braze package.
- 2. Ignition bridge assembly.
- 3. Igniter assembly.
- 4. Braze package assembly.
- 5. Materials handling and process flow.
- 6. Detailed assembly and testing instructions.
- 7. Procedure for calorimetric determination of total heat content of exothermic systems.
- 8. Radiographic inspection film.

To assist in the design and evaluation of the exothermic braze packages many of the units were radiographically inspection. Examination of the resulting films made it possible to determine if all components were structurally sound and properly assembled. All of the final thirty-two (32) braze packages fabricated as flight hardware in Phase III were radiographically inspected as were many of the Phase II packages. A typical film is included as a part of Appendix B.

CONCLUSIONS AND RECOMMENDATIONS

At the completion of Phase I a design had been selected for 0.00635 meter (1/4") ID and 0.01905 meter (3/4") ID exothermic brazing packages. After extensive testing at Marshall Space Flight Center it was decided to modify the design in order to increase relability to the 95% level. In Phase II design of the units was modified to include:

- 1. Rotation of the braze package around the sleeve was prevented.
- 2. Igniter assemblies were anchored firmly parallel to the sleeve and within 0.008128 meter (0.032") of the exotherm material.
- 3. A stainless steel tube housing replaced the aluminum housing.
- 4. Exotherm System #34 replaced System #36.
- 5. Ball and socket ceramic insulators were used as external insulation on the igniter wires to replace the less durable glass sleeving.

With these improvements a 95% level of reliability appears to have been achieved. A standardized manufacturing and inspection procedure (Appendix B) was established for controlling the quality of the exothermic brazing units.

Based upon the fabrication and testing of the 90 units produced under Phase II and Phase III it can be concluded that:

- 1. Reliable exothermic brazing packages can be fabricated for joining 0.01905 meter (3/4") diameter stainless stee1 tubing under hard vacuum (10^{-8} 10^{-6} Torr) at gravity and zero gravity conditions.
- 2. A reliability level of 95% or greater can be achieved in manufacturing these units.
- 3. Brazing units for the joining of tubing sizes other than 0.01905 meter (3/4") diameter appear feasible at least up to 0.1524 meter (6") ID.
- 4. Whittaker Exotherm System #34 is more reliable than System #36 for providing uniform heat distribution under vacuum conditions using a 24-volt power source to ignite the exotherm.
- 5. Two-way alloy flow can be obtained under gravity vacuum conditions.

6. The heat pulse duration of the exothermic packages can be adjusted to prevent or minimize intergranular penetration of the braze alloy into the parent metal.

It is recommended that further work be conducted in the development and application of exothermic brazing units. Areas that should be covered include:

- a. Determine brazing package requirements for stainless steel tubing diameters up to 0.1524 meter (6") or more.
- b. Evaluate feasibility of joining various types of lightweight metal structures by exothermic brazing techniques in space environments.
- c. Investigate use of exothermic units for adhesive bonding and joining of organic materials under controlled environments.
- d. Examine stability of exotherm system #34 in thin sheet form and investigate high speed manufacture of exotherm coupons.
- e. Study manufacturing methods for producing low cost high performance exothermic brazing units.

APPENDIX A

COUPLING DESIGN CALCULATIONS

DESIGN OF 1/4-INCH COUPLING

Material: Stainless steel tubing and couplings of 347 steel tube wall; thickness 0.020 inch.

Tensile strength of 347 (Republic Steel Data Sheet):

Cold worked 100,000 psi to 150,000 psi Annealed 75,000 psi to 100,000 psi

Using the lowest value of annealed condition (as brazed), in Barlow's formula

(A)
$$P = \frac{2 \text{ ST}}{\text{ID}}$$
 $\frac{150,000 (0.020)}{0.210} = 14,300 \text{ psi}$

where

St = 75,000 psi

t = 0.020 inch

ID = 0.250 - 0.040 = 0.210 inch

(B) Minimum braze length necessary to withstand tube burst pressure:

$$x = \frac{PA}{CBM_s}$$
 $\frac{14,300 \times 0.035}{0.785 \times 20,000}$

P = 14,300 (Burst pressure) x = 0.032 inch

 $A = 0.785 (ID)^2$

 $C = 3.1414 \times OD$

 $BM_s = 20,000 \text{ psi (shear strength of braze alloy)}$

Actual coupling braze length safety factor = 8.75

DESIGN OF 3/4-INCH COUPLING

Material: Tubing and couplings of 347 stainless steel; tube wall thickness 0.049 inch

Tensile strength: annealed condition 75,000 psi

(A) Burst pressure

$$P = \frac{2 \text{ St}}{1 \text{ D}} = \frac{2 \times 75,000 \times 0.049}{0.652} = 11,300 \text{ psi}$$

where

(B) Braze length necessary to withstand burst pressure (use 12,000)

$$x = \frac{PA}{CBM_S}$$

$$P = 12,000 \text{ psi}$$

$$A = 0.785 \text{ (ID)}^2 \quad D = 0.652$$

$$C = 3.1414 \times OD \quad OD = 0.750$$

$$\frac{12,000 \text{ (0.334)}}{3.14 \times (0.750) 20,000}$$

$$\frac{0.401}{4.92} = 0.085 \text{ inch}$$

 $BM_s = 20,000 \text{ psi (Braze alloy shear strength, conservative)}$

Actual coupling braze length safety factor = 7.15

APPENDIX B

EXOTHERMIC BRAZE PACKAGE INSPECTION PROCEDURE CONTRACT NAS-8-20829

Introduction:

This procedure was prepared in compliance with modification #8 of Contract NAS-8-20829.

Whittaker Corp., Research and Development Division, is a research and development company primarily engaged in contract research with the responsibility for inspection, reliability testing and certification of all products, processes and fabricated hardware assigned to the individual scientist or engineer.

Whittaker has delivered to NASA reliable exothermic braze packages produced in accordance with the terms and requirements of the contract. To accomplish this requires frequent inspection and testing throughout the entire cycle of fabrication and assembly of the exothermic braze packages. Accordingly, the following inspection procedure has been prepared to control the quality of the exothermic braze units. As changes occurred during the life of the contract that had a significant effect on the provisions set forther in this inspection procedure, the procedure was revised accordingly to reflect those changes.

Components:

Breakdown of components comprising a braze package:

Item <u>Number</u>	Description (quantity)	Processing Procedure	Drawing <u>Number</u>
1	Sleeve Coupling (1)	GFE - Modification	95M10487-3 95M10487-1
. 2	Outer Housing (1)	In-house Fabrication	95M10493
3	Braze Alloy Rings(2)	Out-of-house Fab.	95M10494
4	End Plates (1 + 1)	11	95M10485-3 95M10485-1
5	Exothermic Rings (3)	In-house Fabrication	95M10488
6	Fibered Insulation(N.A.)	In-house Fabrication	95M10288
7	Igniter Housings (2)	11 .	95M10491
8	Igniter External In- sulation (ceramic ball/socket)	Purchase	95M10486

Components: (continued)

Item Number	Description (quantity)	Processing Procedure	Drawing Number
9	Igniter Bridges (2)	In-house Fabrication	95M10490
10	Igniter Insulation Plugs (2)	11	95M10489
11	Igniter Potting Cement	In-house Preparation	95M10486
12	Igniter Retainers (2)	In-house Fabrication	95M10495
13	Igniter Pyrotechnic(NA)	In-house Preparation	95M10486

Braze Package Fabrication Inspection Procedure:

The criteria for acceptance or rejection of the component parts of the braze package will be their conformance to the dimensions and tolerances listed on the appropriate drawing. The instruments used for measurement will be calibrated against a set of gage blocks whose calibration is traceable to the Bureau of Standards as per Whittaker SPI Document #61.41 paragraph 3.3.1.

The assembly procedure below contains the best points which we believe are necessary for delivery of highly reliable braze packages. Package assembly takes place in the following three steps:

- 1. Ignition bridge assembly.
- 2. Igniter assembly.
- 3. Braze package assembly.

Step 1. Ignition bridge assembly.

- (1) Cut leads to length.
 - (2) Cut bridge wire to length.
 - (3) Crimp bridge wire to leads.
- *(4) Resistivity measurement of ignition bridge. Resistivity determined with Wheatstone Bridge.

^{*}Inspection

Step 1. Ignition bridge assembly. (continued)

An acceptable bridge consisting of two 12-inch length #18 (0.040 in.) copper leads and 0.008" tungsten were produced with a resistivity reading in the range of 0.060 ohm to 0.080 ohm. Tungsten wire must be coiled 8 turns on an 0.020" mandrel in order to provide contact to the pyrotechnic 116/17 Ti and uniform ignition.

Step 2. Igniter Assembly

- (1) Insert ignition bridge into ceramic insulator.
- (2) Insert above assembly into igniter housing.
- (3) Inject inorganic cement (Sauereisen #8) into housing above the ceramic insulator to a point below the bridge wire.
- (4) Air dry for 8 hours.
- (5) Dry in oven at 230°F for 6 hours.
- *(6) Test resistivity of ignition bridge. (Same as #4, Step 1)
 - (7) Rivet igniters to end plates.
 - (8) Fill igniters with pyrotechnic ignition mixture (system 116/17T).
- *(9) Test resistivity of ignition bridge. (Same as #4, Step 1)

Step 3. Braze Package Assembly

- (1) Rivet back end plate to cylindrical housing.
- (2) Insert braze alloy rings into coupling.
- (3) Insert coupling into end plate octagonal opening.
- (4) Insert fiberfrax end plate insulation into package.
- (5) Slide exotherm rings over coupling.
- *(6) Measure distance between exotherm ring shoulder and cylindrical housing end. Measurement to be taken by depth gage in insure 0.000 to 0.030 in maximum clearance between exotherm ring and igniter face.
 - (7) Insert fiberfrax radial insulation.
 - (8) Position fiberfrax end insulation over igniters and up against inside face of front end washer.
 - (9) Position front end washer over cylindrical housing keying octagonal opening to coupling flats.

^{*}Inspection

Step 3. Braze Package Assembly (continued)

- (10) Rivet front end washer to cylindrical housing.
- (11) Insert igniter wires into ceramic plug (DWG 95M10489) approximately 1/8" high. Add ball and socket ceramic insulators (The Star Porcelain Co.) 0.110" OD x 0.056" ID x 0.110" long to insulate each wire.
- *(12) Perform resistance measurement on igniters. (Same as #4, Step 1)
- *(13) Perform short circuit test on igniters. Use of VOM to insure that no part of the ignition circuit is in electrical contact with any other part of the package. Reading should show infinite resistance.
- *(14) Take X-radiograph of braze package. The X-radiographs will be made under the following conditions for proper resolution:

Power: 90 KV @ 5 ma
Time: 5 min.
Focal Distance: 48 "
Film: Type M

The unit will be acceptable only if:

- 1. The igniter units lie parallel to the package axis.
- 2. The igniter faces lie within the 0.000 to 0.030 max. clearance range to the exotherm rings.
- 3. The exotherm rings are centered over the coupling.
- 4. The exotherm rings display no cracks or breaks which would cause a part or parts of the ring to assume a position making contact for either complete heat source ignition or thermal transmission to the coupling questionable.

Process Flow:

Metal Components

The new materials used for in-house fabrication of the metal components of the braze unit, namely Items #2, 7 and 11 on the components list, are labeled (material and contract work order number) and stored in a limited access area. These materials are transferred directly to the machine shop facility where they are used to fabricate the components according to the appended manufacturing instructions. After fabrication the materials are transferred to the exotherm processing laboratory which also has limited access for inspection as per the manufacturing instructions.

Those components conforming to manufacturing instruction tolerances are vapor degreased and placed in containers denoting acceptability for braze package assembly.

Those components not confirming to manufacturing instruction tolerances are placed in one of two categories in properly labeled containers:

- 1. Components which may be reworked.
- 2. Components which may not be reworked.

Components in the first category will be subjected to rework and inspection as per the applicable manufacturing instruction. Components in the second category will be held for scrap.

Items listed as #3 and #4 on the components list which are fabricated out of house are subject to the same inspection, categorizing and cleaning procedure as spelled out for items #2, #7 and #11 above.

Igniter Pyrotechnic and Exotherm Heat Source Components:

Materials Draw

The new materials comprising these components are drawn from sealed containers in the stores area, weighed and packaged in labeled polyethylene bags before being transferred to the exotherm processing laboratory. Solvent to be used in the wet blending process is also drawn from the stores area in their original containers.

Blending

The raw materials are wet blended in a ceramic ball mill using ceramic (Al $_2$ 0 $_3$) mixing media. The size of the batch required determines the quantity of solvent and mixing media used. A blending time of 5 hours minimum is used for both components.

After blending, the mixtures are air dried in a fume hood for a minimum of 15 hours. The components are then put through a stainless steel screen and oven dried at 90°C for three hours to remove any residual solvent.

Storage

The dried mixtures are brazed in polyethylene and put up in cans containing dessicant. These cans are labeled with (1) material system type, (2) date of packaging, and (3) work order number corresponding to the contract number.

Testing:

Ignition - Igniter Pyrotechnic

A sample of the ignition pyrotechnic powder is placed in a metal tube with fiberfrax insulation material used for end closure. A thermocouple connected to an X-Y recorder is located in the center of the pyrotechnic charge.

This assembly is thrust into a furnace operating at $1200\,^{\circ}\text{F}$. The point at which the recorded temperature rise slope increases abruptly indicates ignition of the pyrotechnic charge.

Ignition - Exotherm Material

The same procedure is followed as explained in the preceding paragraph with the exception that the furnace is operated at $1800\,^{\circ}\text{F}$.

Calorimetric Output - Exotherm Material

The calorimetric output of the exothermic material is determined by following the procedure spelled out in attached exotherm powder specification D-602. The criteria for acceptance of these materials is conformance to the following limits:

	Igniter Pyrotechnic 116/17Ti	Exotherm System #34
Ignition Temperature	970 ± 50°F	2020 ± 50
Calorimetric Output	too (sa) are ure too our say any	$650 \pm 30 \text{ Cal./g.}$

Acceptable igniter pyrotechnic material is used in the braze units as spelled out in the braze package assembly procedure spelled out above. Rejected material is discarded. Acceptable exothermic heat source material is cold pressed into annular rings as per the manufacturing instructions appended. They are then inspected per the same instructions. Pressed rings are stored in desiccators in the exotherm processing laboratory pending braze package assembly.

Weight

As completed, the individual exothermic braze packages weighed approximately 275 grams each. All units should fall within \pm 10% of this figure unless excessive ceramic ball and socket insulators are used.

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TABLE I

LIST OF MATERIALS FOR
1/4- AND 3/4-INCH BRAZE PACKAGES (Phase I)

Components	Materials Used
Tubing	1/4-inch OD x 0.020-inch Wall 347 Stainless Steel 3/4-inch OD x 0.049-inch Wall 347 Stainless Steel 1-1/4-inch OD x 0.020-inch Wall 2024 T3 Aluminum 2-3/4-inch OD x 0.028-inch Wall 6061 - 0 Aluminum 7/32-inch OD x 0.010-inch Wall 321 Annealed Stainless Steel
Couplings	347 Stainless Steel
Alloy Rings	AWS A5.8-62 Class B Ag-8A BT Lithobraze Handy & Harman
Wire	Tungsten 0.008-inch Diameter Grade CS General Electric Copper #18 Soft Drawn Mil-W-3861/5 QQW-343/5 Alpha Wire Corporation
Exotherm	Rings - Whittaker #36 Igniters - Whittaker #116 and #17
Insulation	Fiberfrax Blanket - 1/2-inch Thick LO-CON 6# Density - Carborundum Fiberfrax Felt - 1/8-inch Thick (970-18 Paper White) Carborundum Ceramic (Igniters) Electrotemp Cement No. 8 Powder - Sauereisen Cements Co.
Washers (package end)	0.020-inch Type 302 Annealed Stainless Steel (1/4-inch size) 0.025 Type 302 Annealed Stainless Steel (3/4-inch size)
Washers	#12 Stainless Steel or Brass (1/2-inch OD X 7/32-inch ID X 0.040 inch)

NARMCO RESEARCH & DEVELOPMENT A Division of Telecomputing Corporation 3540 Aero Court San Diego 23, California

EXOTHERM POWDER	S P E C I F I C A T I O N
Type: <u>Test Procedure</u>	No. <u>D-602</u>
Title: PROCEDURE FOR CALORIMETRIC	Date: 8 May 1964
DETERMINATION OF TOTAL HEAT CONTENT	Supersedes No.
OF EXOTHERMIC SYSTEMS	Dated:
Prepared By: 7. J. N. La Dilas	Obsoletes No.
Approved By: S. Rodney	Dated:
Project Officer: F.J. Filippi	
Department Head: R. A. Long	
Technical Director: frank F. J. Riel	
ii	·

PURPOSE:

To establish a standard calorimetric test procedure for exothermic systems.

SCOPE:

Limited to calorimetric test procedure applicable to exothermic powders. This specification includes procedure for calibration of calorimeter; it does not include calorimetric performance limit values.

APPLICABILITY:

Exotherm systems #213 Reagent, #213B, #126, #32, #34.

MATERIALS, SUPPLIES, AND EQUIPMENT

- 1. 1401 Calorimeter (Parr)
- 2. 1601 Thermometer (Readable to 0.05°F (Parr calibrated)
- 3. 3003 Thermometer Reading Lens (Parr)
- 4. 2ACN Cup 22 ml, 95% Nickel-Fusion (Parr)
- 5. AIADC-Bomb Assembly (Ref. Parr Catalogue 61-1)
- 6. 0.008-in. dia. Fuse Wire, Tungsten (G.E. 218 or equivalent)
- 7. Burgess Dry Cell, 6-volt, 4F4H
- 8. 2000 ml Volumetric Flask, or equivalent
- 9. Coors Crucible #230-000, C.B. 199
- 10. Calorimeter Leads, or equivalent
- 11. Weston D.C. Ammeter, scale 0-10 amps, D.C. #2621010
- 12. Clebar Stopwatch, Accurate to 1/5 second
- 13. Distilled Water 2000 ml source

Spec. No. <u>D-602</u> 8 May 1964 Page Two

- 14. Balance, accurate to 1/10 of 1 mg (Christian Becker)
- 15. 20AC2 Stirring Clips, Two (Parr)
- 16. Thermometer Correction Sheet
- 17. #21AC Wrench (Parr)
- 18. #22AC2 Bench Socket
- 19. #AllAC Deflector Tube (Parr)
- 20. #A5C Can, Water (Ref. Parr Catalogue #61-1)
- 21. Standard Benzoic Acid Powder (Parr)
- 22. Potassium Perchlorate (Parr)
- 23. Sodium Peroxide with dipper provided
- 24. Standard calculation' sheet

STANDARDIZATION OF THE CALORIMETER

Procedure:

(Reference: Peroxide Bomb Calorimetry. Parr Manual 122)

- 1. Set up empty calorimeter, with water container removed, cover in place with thermometer inserted so that the jacket comes to thermal equilibrium.
- 2. Attach tungsten ignition wire, cut to 5-cm length ± 0.5 cm, to electrodes on bomb head.
- 3. Add 0.500 g \pm 0.5 mg of standard benzoic acid to 1.000 g \pm 0.5 mg of potassium perchlorate accelerator in the fusion cup.
- 4. Mix vigorously with stirring rod for 5 minutes until benzoic acid is completely powdered and mixed with accelerator.
- 5. Add one dipper of sodium peroxide and stir until the benzoic acid-potassium perchlorate mixture is uniformly distributed throughout.
- 6. Any material adhering to stirring rod should be brushed into the cup. Add porcelain crucible.
- 7. Close and seal the bomb. Tighten screwhead using the wrench, while holding bomb in bench socket.
- 8. Attach #20AC stirring blades to bell body of bomb with blades downward and space so that holes in bell body are not obstructed.
- 9. Set deflector tube in water container, add 2 liters distilled water, after adjusting its temperature 1.5°F to 2°F below that of the room.
- 10. Read and record on standard calculation sheet the initial jacket temperature, and the temperature of the calorimeter chamber empty.
- 11. Assemble the calorimeter for test.
- 12. With bomb rotating, allow a period of 5 minutes for the temperature to become equalized before igniting the charge.

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- 13. Record the water temperature, apply ignition power for 6 seconds, and take temperature readings at ½ minute intervals. Monitor the time with a stopwatch.
- 14. After reaching maximum temperature, record elapsed time for the period of total rise. (p. 26 Parr Manual)
- 15. Apply the appropriate corrections for thermometer error and radiation, and subtract corrections for accelerator (0.200°F, and hydration 0.185°F), to obtain net corrected temperature rise. Use this to compute the water equivalent.
- 16. Water equivalent definition that amount of calories necessary to raise the temperature of the calorimeter system 1°C.
- 17. The calorific value is an <u>adjusted</u> water equivalent to be used when the calorimeter is used to determine hydrocarbon fuel heat values. The water equivalent is 68.5% of the calorific value. Since this system is standardized at <u>calorific value</u> 3050 (average of 4 runs), the water equivalent is 68.5% of 3050 or 2090. (Note: Standardization materials are hydrocarbons.)

TEST PROCEDURE:

- 1. Set up calorimeter, with the water container removed. Set the cover in place and insert the thermometer in the empty calorimeter chamber.
- 2. Attach the tungsten ignition wire to the bomb head.
- 3. Tare the crucible and weigh out the exact amount of exotherm to be tested into it. Weight is determined to ± 0.1 mg. The exotherm charge should be between 3 and 4 g.
- 4. Place crucible containing exotherm into fusion cup #2ACN.
- 5. Place fusion cup into bomb assembly, assemble bomb head, tighten screwcap and attach stirring clips; be certain floating bottom is in place and right side up in lower end of bell body.
- 6. Tighten screwcap firmly with wrench while holding bomb in bench socket.
- 7. Attach stirring blades to bell body of bomb with blades downward, and space so that holes in bell body are not obstructed.
- 8. Set deflector tube in water container, add 2 liters of distilled water, after adjusting its temperature to 1.5 g to 2°F below that of the room.
- 9. Read and record on a standard calculation sheet the initial jacket temperature, and the temperature of the empty calorimeter chamber.
- 10. Assemble the calorimeter for test.

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- 11. With the bomb rotating, allow a period of 5 minutes for the temperature to become equalized before igniting the charge.
- 12. Record the water temperature, ignite the charge and take temperature readings at }-minute intervals; apply ignition power for 6 seconds with stopwatch.
- 13. After reaching maximum temperature, record elapsed time of period of total rise.
- 14. Obtain radiation correction.
- 15. Compute the results by converting the corrected temperature (°F) rise to degrees centigrade, and multiplying this by 2090, the water equivalent of the calorimeter system. This will yield the total calorie output contributed by the exothermic reaction and the ignition energy.
- 16. Ignition wire correction calculation:
 6 seconds x 6 volts x 4.5 amps = 162 watt-seconds.

Heat output = $\frac{162 \text{ watt-second}}{3600 \frac{\text{watt-second}}{\text{watt hour}}} \times 860 \frac{\text{cal}}{\text{watt hour}} = 39 \text{ cal}$

Subtract ignition wire correction from total calorie output.

17. Divide calorie output by weight of exotherm sample in grams to obtain unit heat output.

Unit heat output = (Total calories) - (Ignition calories)
Sample weight, grams

18. Record unit heat output on the standard calculation sheet, and enter in log book.